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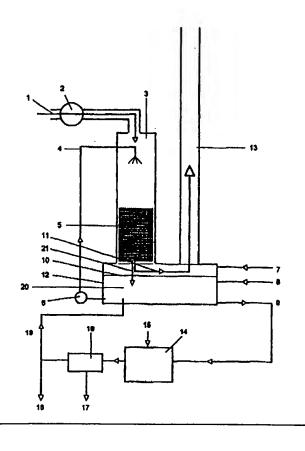
# INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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### (54) Title: A METHOD FOR REMOVING MERCURY AND SULPHUR DIOXIDE FROM GASES

#### (57) Abstract

A gas washing water consisting of sea water provided with sodium hypochlorite to give a redox potential in the range 600-800 mV is fed co-currently with a flue gas containing mercury and  $SO_2$  gas downwards through a bed of limestone to a collection tank beneath the limestone bed. Even if the oxidation of the  $SO_2$  gas to give sulphuric acid, the pH of the gas washing water will remain within 5-6 units, and 90-93 % of the total amount of mercury in the flue gas from the foundry is removed after a residence time of only 0.05-0.1 second in the limestone bed. The gas washing water is purified with respect to mercury by a common sulphide precipitation, and precipitated gypsum in the gas washer will act as an in situ filter aid for the minute sulphide particles, such that the subsequent filtration can easily be carried out. The purified gas washing water contains less than  $1 \mu g Hg/liter$ .



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# A method for removing mercury and sulphur dioxide from gases

The present application refers to an improved process and 5 device for the removal of mercury and sulphur dioxide from flue gases from, for example, foundries, power stations, refuse disposal plants, and crematories, by washing the gas with chloride containing water, such as sea water, to which 10 is added an oxidizing agent which oxidizes all of the mercury to Hg(II) ions, and by using such high redox value in the washing water that Hg \* salts cannot precipitate. The gas and the washing water are passed co-currently down through a bed of consumable limestone. The mercury(II) ions 15 will then dissolve easily in the chloride containing washing water, as a chloride complex, and the oxidized sulphur oxide is converted to sulphuric acid being neutralized by the consumable limestone, such that the pH value of the washing water remains almost constant without the need for 20 using an aqueous solution of sodium hydroxide as a neutralizing agent. Further, the bed of limestone will be functioning as a turbulent mixer of gas and washing water for a rapid oxidation, and since the limestone is slowly consumed, deposits such as gypsum and dust will continuously 25 be washed away from the limestone gravels without clogging the bed.

The present process is carried out by adding to the gas washing water containing 30-40 g chlorides/liter an oxidizing agent, such as sodium hypochlorite, until the redox potensial of the water is within the range 600-800 mV. The gas washing water and the flue gas are fed co-currently down through the lime stone bed, in which they are mixed intimately for a rapid reaction and absorption of the mercury in the aqueous phase.

As flue gases also often contain SO<sub>2</sub> gases, sulphuric acid

is formed by the acidation which reduses the pH value of the gas washing liquid.

According to the invention, the gas is passed co-currently with the gas washing water through the bed of, for example, limestone. The limestone will neutralize the acid(s), and hence the pH of the gas washing water will remain nearly constant in the range 5-6 pH units, and thus completely eliminate the need for the use of aqueous sodium hydroxide, as well as the possibility of SO<sub>2</sub> reducing the oxidized mercury to Hg' salts which can precipitate or to metallic Hg which is reintroduced into the flue gas.

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The bed of consumable limestone is constantly washed with
the gas washing liquid, so that dust and formed sulfates,
such as gypsum and other particles, are continuously washed
into a collection tank arranged below the gas washing
tower, from which the washing water is conducted to a
adjacent water purification plant in which the mercury is
removed as sulfide by common precipitation.

Gypsum particles and other particles act as an *in situ* filter aid for the minute precipitated mercury sulphide particles in the water purification plant, thus easing the separation of mud and water, for instance by filtration, and the chloride water, which will be practically devoid of mercury (<1  $\mu$ g Hg/l), can either be disposed to a recipient or returned to the gas washer.

In addition, it is preferred to conduct the gas and the gas washing water co-currently down through the bed of limestone and into the collection tank for the gass washing water, in which tank the gas washing water and the gas phase meet the surface of the water in the tank. The water droplets in the gas phase will then be absorbed on the water surface by adjusting the distance between the bed of limestone and the water surface in the collection tank for

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the gas washing water as well as the length of said surface, before purified gas is exhausted to a chimney. This will eliminate the need for complicated drip-catchers, being in common use in counter current gas washing.

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It is known that the mercury in the flue gases exists both as oxidized mercury and metallic mercury vapour. The metallic mercury vapour is the most difficult to remove with wet washing of the flue gas, as the vapour must be oxidized before it can be absorbed in the gas washing water.

Flue gases from foundries and power stations have a relatively even content of mercury and other gases, such as  $SO_2$ , which reduces the mercury to metallic vapour in the flue gas, which can contain from 10-20  $\mu$ g Hg/Nm³ up to 300-500  $\mu$ g Hg/Nm³.

The smoke gases from refuse disposal plants and crematories show large variations in the consentration of mercury. The consentrations can be 100-10.000  $\mu g/Nm^3$  gas over a short period of time, 5-10 minutes, due to sources containing large amounts of mercury, such as dental amalgam.

A wet wash process for effective removal of mercury hence must have very quick reaction kinetics to be able to handle these relatively short and intense peaks of mercury in the flue gases.

In accordance with the invention, it is preferred to add sodium hypochlorite to a gas washing water based on sea water, such that the redox potential of the sea water remains in the range 600-800 mV, and subsequenty feed the gas and the gas washing water down through a bed of limestone, such that the pH in the gas washing water stays between 4 and 6 pH units, and subsequently into a tank in which particles and the gas washing water are collected, whereafter the gas washing water is purified in an adjacent

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water purification plant, and one part of the purified water is returned to the gas scrubber and one part is led to a recipient. This process removed 90-93% of the mercury from the fluid gas from a foundry at a residence time in the gas scrubber of only 0,05-0,10 seconds.

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The purified flue gas contains 11-16  $\mu g$  Hg/Nm³ after purification.

The present invention is a simplification and improvement of known methods, which is in demand with the increasing concern about mercury pollution, in particular from refuse disposal plants and crematories, but also from foundries and power stations.

Oxidation of mercury with absorption in a chloride containing gas scrubber water has been described in numerous publications and patents, inter alia US patent 5.009.871.

US 5.009.871 teaches that reducing gases such as SO<sub>2</sub> gas reduces oxidized Hg to metallic Hg, which leaves the gas washing water and is reintroduced into the flue gases. In order to prevent this, US 5.009.871 teaches that the redox in the gas washing water must be >300 mV, and preferably in the range 530-710 mV for approximately 80% reduction of the Hg contents in the flue gases.

Further, US 5.009.871 teaches that due to hydrochloric acid and other reducing gases in the flue gas from incinerators, the addition of, for example, NaOH to the gas washing water is necessary in order to give a pH of 8.

It has surprisingly been found that the teaching of US 5.009.871 concerning the addition of caustic (NaOH) to increase the pH to approximately 8 in the gas washing water is not necessary when the gas washing water an the gas is led co-currently down through a bed of limestone.

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A person within the state of the art will know that at pH values of 8 in the gas washing water, carbonates are formed when CO<sub>2</sub> is present in the gases. The produced carbonate can easily be deposited on the surface of the gas scrubber, in valves, tubes, and in tanks, and hence a pH of 8 in the gas washing water may be undesirable when the gas contains CO<sub>2</sub> gas.

Carbonate formation decreases at pH values of < 6 in the gas washing water and is completely eliminated at pH < 4.

In accordance with the invention, the pH in the gas washing water consisting of sea water is always in the range 5-6 units, and no formation of carbonates was observed, whereas it was observed when caustic was used at pH values of 8.

This could in no way be deduced from US 5.009.871, which describes an apparatus which bubbles a gas through an aqueous solution at the rate of 1 l/min (column 8, lines 1-10).

US 3.849.267 teaches that if chlorine gas is added to a gas containing mercury before the gas mixture is fed to beds consisting of, respectively, a 30 cm thick bed of 2 inch saddle formed fillings of a ceramic material, then through a 25 cm thick bed of 1 inch saddle formed fillings, then a 5 cm bed of glass wool of 4  $\mu$ m fibres, and finally through a 50 cm thick bed of granulated active carbon (column 4, example 5), 20-30 mg Hg/m³ to 0,02 mg Hg/m³ (= 20  $\mu$ g Hg/m³) of mercury will be removed from the gas.

The present invention needs only a bed of limestone, and no bed of active carbon is necessary to obtain an exhaust gas containing 10-15  $\mu$ g Hg/m³, something which cannot be deduced from US 3.849.267, as the examples 1-4 of US 3.849.267 teach that without active carbon, the contents

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of mercury in the purified gas is 100, 50, 100-200 and 80  $\mu$ g Hg/m³, respectively.

Further, US 3.849.267 teaches that mercury is deposited on the solid bodies in the gas scrubber (claim 1), and it is mentioned that the deposits are in the form of Hg<sub>2</sub>Cl<sub>2</sub> (column 1, lines 46-51, and column 5, lines 5-14).

This is contrary to the teaching of the present invention, in which it is mandantory that all mercury is continuously oxidized to Hg\*\* and absorbed in the washing water, and is not deposited in the limestone bed.

Norwegian patent No. 148.962 refers to a method in which mercury is removed from a flue gas containing significant amounts of SO<sub>2</sub> after rosting of sulfide ores, in order to produce a sulphuric acid with a low content of mercury.

Removal of mercury from the gas takes place by using a washing solution containing 0,02-20 g Hg\*\*/liter.

This washing solution oxidizes the metallic mercury in the gass to  $Hg_2Cl_2$ , which is subsequenty removed in an adjacent plant.

On page 7, lines 14-16, it is mentioned that if the contents of mercury(II) ions in the solution (= washing solution) becomes too low, the mercury can no longer be taken up by the solution. On the same side, line 19, it is mentioned that said mercury contents preferably should be between 10 and 50 mmol/l (= 2-10 g Hg/l).

In accordance with the invention, no  $Hg_2Cl_2$  is produced, as all the mercury in the flue gas is oxidized to  $H^{**}$  ions and is absorbed by the washing water as a chloride complex. This implies that the washing water in accordance with the invention in reality does not necessarily have to contain

mercury, even if in practice the contents will be 1-5 mg Hg\*\* ions, or approximately 1/1000 of what is stated in NO 148.962, and even then 90-93% of the mercury, in accordance with the present invention, will be removed with a residence time in the gas scrubber of only 0,05-0,10 seconds.

At the same time all SO<sub>2</sub> is removed, which is not the case for NO 148.962, operating with an acid washing solution.

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10 Norwegian patent NO 154.682 refers to a process for the purification of gases for metallic mercury by precipitation of Hg<sub>2</sub>Cl<sub>2</sub> with a washing water containing 0,02-60 g Hg<sup>++</sup>/liter. The essential part pf NO 154.682 is that the gas is moistened to saturation at 60-70°C and then quench the 15 washing water sufficiently to cool the gas to a temperature at which the vapour in the gas condenses and thereby removes mercury containing liquid (claim 1).

In accordance with the invention, there is no need for such cooling of the gas as taught by NO 154.682, as the gas and the washing water is sent co-currently through the bed of limestone and is imping on the surface of the washing water in the tank for washing water, where the descending washing water and water droplets in the gas phase are absorbed.

25 This is a preferred embodiment of the present invention.

The method of the invention operates according to a principle contrary to the teachings of NO 154.682, which operates according to the counter-current principle, in which the washing water decends and the gas ascends. As known to the person within the state of the art, the counter-current principle produces particles in the gas, and hence cooling of the gas or an efficient drop catcher is essential when the washing water contains up to 2-10 g mercury/liter.

EP 0.709.126 A1 discloses a method and a device for the

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continuous removal and precipitaton of mercury from gases. The washing solution in accordance with example 2 contains 8 g Hg(II)/l, 13 g/l SCN $^-$ , 10 g/l sulphuric acid and 2 g/l active carbon.  $\rm H_2S$  gas is added to the gas to be purified, which is not necessary according to the instant invention.

The device used in EP 0.709.126 Al is a counter-current scrubber with a drop catcher after the reactor, which in turn is filled with "filler bodies", which must be understood as being inert (=insoluble) in the solution and gas flowing through the reactor.

In accordance with the invention, both mercury, in the form of a Hg(II) complex, and the SO<sub>2</sub> gas in the flue gas, in the form of sulpuric acid, are washed away by using sea water to which an oxidizing agent is added, then the gas and the washing water are passed counter-currently down through a bed of consumable carbonates in which the sulphuric acid is neutralized, and the washing water with the absorbed mercury, as well as solid particles, pass through the carbonate bed and into the tank for washing water beneath the carbonate bed, then imping on the surface of the washing water, and thus the water droplets in the gas phase are absorbed without the need for a subsequent drop catcher. This is the preferred mode and device for carrying out the present invention.

The invention represents a greatly simplified process and device for removing mercury and SO<sub>2</sub> from flue gases, and we are of the opinion that this combination of method and device for carrying out the method cannot be deduced from the above mentioned patents/applications or literature and other publicly accessible publications known to the applicant.

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Another method for carrying out the invention, instead of adding sodium hypochlorite to the gas washing water, is to

pass an electric current through the chloride containing sea water. This produces chlorine gas, which keeps the redox potential at the desired level. Other oxidizing agents can also be used, such as peroxides and persulphates.

It is also within the scope of the invention to utilize other carbonates than limestone (calcit), such as dolomite, chalk, shell sand, and magnetite.

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It is a requirement that the carbonate medium has a low solubility in hot water, such as limestone (calcit) with a solubility of approximately 0,02 g/l water, og magnesium carbonate (magnecite) with a solubility of approximately 0,1 g/l, whereas dolomite has a solubility of approximately 0,32 g/l hot water.

The present invention will now be described with reference to figure 1, where (1) indicates the gas to be purified and 20 (2) is a fan device transporting the gas (1) into the washing chamber (3), in which the gas is washed with a washing solution (4) provided from the collection tank (12) via a pump (6), and in which the gas (1) and the washing solution (4) descend co-currently through a limestone bed 25 (5) to exit together at the top of the tank (12), and where the washed gas (11) is transported along the surface (10) of the washing water (20) in the collection tank (12) and exhausted through a chimney (13), and the used washing solution (21) is collected in the tank (12) together with 30 the washing water (20), in which tank an oxidizing solution (7) and a chloride solution (8) is added; the excess water (9) is led to a water purification plant (14) in which a sulphide solution (15) is added, whereafter the water and the precipitated solid particles are separated in (16), 35 where the sludge (17) is removed, and a part of the purified chloride water is fed to a recipient (18) and the remaining part (19) is then returned to the tank (12).

The invention will be further described by the following examples.

#### 5 Example 1

In this example the redox potensials at which SO<sub>2</sub> is oxidized to sulpuric acid at different pH values was established.

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This was done by adding a fixed amount of NaHSO<sub>3</sub> to sea water with a known pH value. Subsequently, small amounts of potassium iodide (KJ)/starch solution were added as an indicator for titration of NaHSO<sub>3</sub> using NaClO. When a blue colour developed, the SO<sub>3</sub> was consumed, and the redox and pH were recorded. This is a common iodine titration of chloride/hypochlorit in water.

Table 1 Results of oxidation of SO<sub>2</sub> with NaClO

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Нф	Redox value (mV) at which a blue colour was noted
3,0 4,4 6,0 7,0 8,0 9,5	340 322 In accordance with the invention 290 In accordance with the invention 265 235

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#### Comments

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The results show that at redox values > ca. 300 mV at pH values in the range 3-6 mercury cannot be reduced by the  $SO_2$ .

# Example 2

A flue gas from a melting furnace was drawn through a very efficient venturi scrubber, consisting of a laboratory water jet vacuum pump.

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The washing water was pumped from a collection tank for washing water by means of a pressure pump feeding the water jet pump, and hence sucking the flue gas therethrough.

In this manner a very efficient mixing of the gas and the washing water was obtained, and the progress could be observed continuously by taking gas samples both before and after the gas washer, as well as water samples from the washing water. Samples were taken at the beginning and the end of a gas measurement.

Caustic (NaOH) was used to keep pH at a constant value.

#### 20 <u>Table 2</u> <u>Results, mass balance</u>

Test	Anal		the w	ash water	Total amount $\mu$ g Hg. Absorbed in the washing water as:		
	Start	Stop	Hq	Redox	Hg++	Hg <sup>+</sup>	
1	9,7	15	8,0	750	121	0	
2	4,6	33	6,3	800	631	0	
3	54	48	5,9	600	0	135	
4	55	38	5,9	550-600	0	435	
5	0	0,24	5,2	200-300	5	12	

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#### Comments

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1 and 2 show that all of the mercury was absorbed in the sea water as Hg\*\*.

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The tests 3 and 4 show that Hg is precipitated as Hg $^{\star}$  at redox values  $\leq$  600 mV. This is indicated by the fact that the contents of Hg in the washing water declined during the test period.

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Test 5 was carried out with pure sea water without addition ofcaustic or hypochlorit. Hg\*\* in the water might be due to dissolution of previously precipitated Hg\* salts.

#### 15 Conclusion

In order to absorb all of the mercury in sea water as  $Hg^{**}$  ions, the redox potential in the sea water must be >600 mV, at pH values in the range 6-8.

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#### Example 3

The flue gas from a melting furnace was burned in a steam kettle to:  $CO_2$ ,  $SO_2/SO_3$  and  $H_2O$ . This gas contained 100-200  $\mu g$  of  $Hg/Nm^3$ .

A gas purification plant which could purify up to  $1000~\text{m}^3$  gas/hour was used, and connected to a water purification plant as shown in figure 1.

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Gas washing liquid: sea water
pH kept constant by means of limestone
Redox adjusted with NaClO, 15% commercial product
Recirculation of the washing water in respect to qas:

 $1 \, l/m^3 \, gas$ 

Addition of sea water: 60 1/hour.

Table 3 Gas purification results

No.	Gas rate	Gas was	hing liqui	d Mercury	contents in gas	$(\mu g/Nm^3)$
NO.	Nm <sup>3</sup> /t	pН	Redox	Inlet	Purified gas	% purification
1	350	5,8	700-850	176	13	93
2	350	6,2	700-850	168	11	93
3	350	5,9	600-800	162	11	93
4	850	5,7	700-800	164	16	90

#### Comments

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No carbonates were formed, and the operation was very stable, due to the stable pH value of the gas washibg water.

In test 4, redox was varied between 600 and 800 mV to 20 simulate variation in process conditions. This did not have any effect on the level of purification.

With a gas rate of 850 Nm<sup>3</sup>/h the linear gas velocity through the limestone bed was calculated at 7 m/sec, and at a recidence time of only 0,055 seconds.

Even this was sufficient for purification, which shows that the instant process is very rapid and effective.

- A mass balance of the distribution of the mercury in test 2, between the gas phase and the gas washing water, showed that close to 100% of the absorbed mercury was absorbed as Hg\*\* in the gas washing water.
- 35 The mercury was removed from the gas washing water by a ususal sulfide precipitation in a subsequent water purification plant. This was effected by adding  $1,3 \text{ l/m}^3$  washing

water from a solution of sodium sulfide (10 g/l, Na<sub>2</sub>S) to the gas washing water in a stirred tank until the redox potential of the water was between -50 and -100 mV. Subsequently, the water and sludge were separated in a filter press. The filtrate contained < 1  $\mu$ g Hg/l. The gypsym formed, and other particles, functioned so well as a filter aid that the filtering resistance in the filter press was < 0.5 bar.

#### 10 Example 4

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This example is a summary of results, showing the efficiency of the instant invention compared to venturi gas washers with and without a drop catcher.

Tabell 4 Results

Gas	washi	ing wate	er	μg Hg/	Nm³
Gas washer type	рĦ	Redox	Raw gas	Purified gas	condence water
Small venturi <sup>1</sup> without drop catcher	5,8	600	74	9,0	63
Big venturi <sup>2</sup> with drop catcher	5,0 4,4	690 790	154 127	52 75	1,1 0,8
Present 3 invention	5,8 5,7	830 640	167 165	15 16	0,6 0,9

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#### Comments

- The venturi washer referred to in example 2, without drop catcher. Very efficient mixing of gas and washing liquid.
- 2): Large venturi washer with a capacity of treating 1000 m³/hour, with drop catcher. Inferior mixing of gas and washing liquid.
  - 3): Gas washer in accordance with the invention with a limestone bed with a cpacity of 850 m³/hour. Very efficient mixing of gas and washing liquid.

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Condense water\*) was collected in an extra bottle before the gas analysis of the purified gas. The mercury contents of the condensed water was calculated to give the stated contribution of Hg in the gas if the condense water had been introduced into the purified gas without a drop catcher.

Further, it can be observed that the effect of a drop catcher in 2) and the effect in accordance with the invention 3), where the amount of condense water in the gas is so small that the purified gas is not polluted.

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It can also be seen that the total mercury purification
efficiency of the venturi washer 2) with the drop catcher
is substantially lower than the purification efficiency in
accordance with the invention 3), when both were tested on
the same gas from a foundry.

#### CLAIMS:

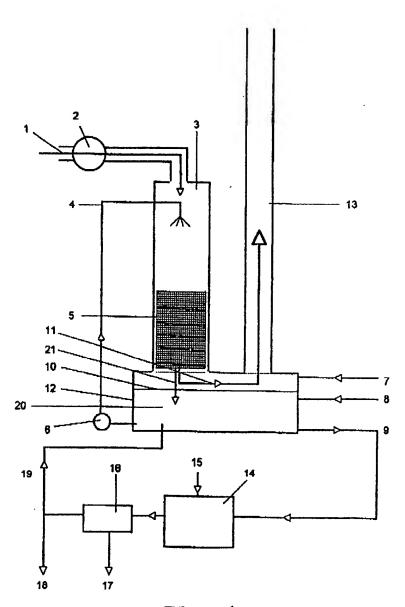
- A method for removal of mercury and sulphur dioxide from gases by an oxidizing wet washing of the gas, oxidiz-5 ing the mercury to Hg\*\* ions and the SO2 gas to sulphuric acid by using a chloride containing aqueous liquid, containing at least 1 g chlorides/l and having pH values in the range 4-8 pH units and redox values in the range 600-10 800 mV, and using one or more alkaline earth carbonates, characterized in that the oxidizing chloride containing washing water and the gas are fed co-currently down through a stationary bed of solid bodies of alkaline earth carbonates in a one-step gas washer into a tank 15 underneath said gas washer, wherein the gas and the washing water is divided, such that the oxidized SO2 gas, which together with the washing water forms sulphuric acid, is neutralized by said carbonate and forms a sulphate, which is continuously washed from the solid bodies of carbonates, 20 thereby exposing new and active carbonate surfaces for further consumption, and in which the oxidized mercury at the same time is absorbed in the washing water as Hg(II) chloride complexes.
- 25 2. A method in accordance with claim 1, c h a r a c t e r i z e d i n that said solid and stationary alkaline earth carbonate is limestone.
  - 3. A method in accordance with claim 1.
- 30 characterized in that the oxidation of the gas washing water preferably is effected by adding sodium hypoclorite.
  - 4. A method in accordance with claim 1,
- 35 characterized in that the cloride containing gas washing water preferably is sea water.

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(12).

Device for carrying out the process in accordance with the claims 1-4, comprising a gas washing tower (3) with an inlet conduit (1) for gas and an inlet conduit (4) for gas washing water, a tank (12) provided underneath the gas washer (3), a purification plant (14) for purification of 5 the gas washing water, characterized in that the inlet conduit (1) for gas and the inlet conduit (4) for activated gas washing water are provided on top of the gas washer (3), and that 10 said gas washer (3) is provided with a bed of alkaline earth carbonates (5) near the bottom of the gas washer (3), and that underneath the gas washer (3) a tank (12) is provided, said tank (12) is provided with an outlet for consumed gas washing water (9) to a purification plant (14) 15 for gas washing water, an outlet (6) for recirculating reactivated gas washing water (4) to the top of the gas washer (3); the tank (12) has inlets for the addition of water (8) and an oxidizing agent (7) as well as outlets for recycling of purified gas washing water (19) to the tank



F1G. 1

#### INTERNATIONAL SEARCH REPORT

International application No. PCT/NO 97/00274

		PCI/NU 9	//002/4
A. CLASS	SIFICATION OF SUBJECT MATTER		
IPC6: E	301D 53/64, B01D 53/50, B01D 53/76 o International Patent Classification (IPC) or to both n	B ational classification and IPC	
	OS SEARCHED		
Minimum d	ocumentation searched (classification system followed b	y classification symbols)	
IPC6: E			
Documental	tion searched other than minimum documentation to the	extent that such documents are include	ed in the fields searched
SE,DK,F	FI,NO classes as above		
Electronic d	ata base consulted during the international search (name	e of data base and, where practicable, s	earch terms used)
WPI,EPO	DDOC		
	MENTS CONSIDERED TO BE RELEVANT		***
Category*	Citation of document, with indication, where ap	propriate, of the relevant passages	Relevant to claim No.
A	EP 0706815 A2 (FORSCHUNGSZENTRU 17 April 1996 (17.04.96)	M KARLSRUHE GMBH),	1-5
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"B" erlier do "L" documen	current but published on or after the international filing date at which may throw doubts on priority claim(s) or which is establish the publication date of another citation or other	"X" document of particular relevance considered novel or cannot be co step when the document is taken	nsidered to involve an inventive
special r	reason (as specified) nt referring to an oral disclosure, use, exhibition or other	"Y" document of particular relevance considered to involve an inventive combined with one or more other	the claimed invention cannot be a step when the document is such documents, such combination
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13 Janu	eary 1997	06 -	02- 1998
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-	S-102 42 STOCKHOLM	Britt-Marie Lundell	<b>.</b>

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